PB-217 677

PROPOSED STANDARD PROCEDURES FOR DETERMINATION OF THE EFFECTIVE SPECIFIC GRAVITY, BULK DENSITY, AND FILTER CAKE RESISTANCE OF PRECOAT FILTER AIDS

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15 June 1968

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FINAL REPORT
PART IV OF VI PARTS
PROPOSED STANDARD PROCEDURES FOR

DETERMINATION OF THE EFFECTIVE SPECIFIC GRAVITY, BULK DENSITY, AND FILTER CAKE RESISTANCE OF PRECOAT FILTER AIDS

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Submitted to:
U. S. Army Engineer Research and Development Laboratories
(Contract DAAK 02-67-C-0408) Ft. Belvoir, Virginia

June 15, 1968

SANITARY ENGINEERING SECTION
ENGINEERING RESEARCH INSTITUTE
IOWA STATE UNIVERSITY AMES

ERI - 263
Project No. 684-S

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DETERMINATION OF THE EFFECTIVE SPECIFIC GRAVITY, BULK DENSITY AND FILTER CAKE RESISTANCE OF PRECOAT FILTER AIDS

INTRODUCTION

Diatomite and perlite filter aids are used to increase the filtrability of water that contains suspended solids. Filter cakes containing the appropriate grade of either diatomite or perlite will entrap suspended solids too small to be removed by sand filters and will accomplish this removal without building up an excessively high headloss.

In considering the purchase of filter aids, two or more may be assumed equivalent when they provide the same quality of filtered water with the same filter cake resistance. The quality of water produced by the different types and grades of filter aids available is influenced by the characteristics of the suspended solids to be removed and by the characteristics of the filter aid used in filtration. The quality of the filtered water improves with the use of finer and finer grades of filter aid, but the filter cake resistance increases when equal weights of the finer filter aids are used as body feed. In practice, it is customary to use the coarsest grade of filter aid that will provide the desired filtered water quality and to adjust the body feed to obtain the desired level of filter cake resistance.

Once a grade of filter aid is selected for a given filtration application, it is frequently necessary to establish its characteristics so that a purchase specification can be written. The specification should be written so that other grades of the same or different materials may be considered for purchase if they provide "equivalent performance." Thus, "equivalent performance" implies that filtration with the equivalent filter aids provides the same quality of filtered water with the same filter cake resistance. Such performance with the same type of material,

for example, can be obtained by using finer grades of filter aid by increasing the rate of body feed. These finer grades of a filter aid are usually slightly cheaper than the coarser grades. The use of increased amounts of finer grades as body feed may provide "equivalent performance" if the cost of purchasing and shipping a greater amount of material to the point of use is not excessive.

The specification adopted may be either a performance specification or a material specification. The performance specification delineates the conditions under which the material purchased will be used and sets forth the results that are expected. The test or whether a material meets the performance specification is whether it produces the desired quality of water with the specified rate of headloss buildup or less at the point of use under actual operating conditions.

The material specification describes the material desired in terms of physical properties needed to achieve the performance desired. Standardized laboratory tests can be used to determine whether the material in question has the specified properties. One of the problems inherent in the use of a material specification lies in determining how much of a finer filter aid is required to provide resistance equivalent to that of a given amount of coarser filter aid, so that the cost of using the finer material can be compared with that of the coarser material. The physical properties described in a material specification must be measureable with enough accuracy that the expected performance of the filter aid can be predicted with accuracy.

In any event, it is necessary that the buyer must know or be able to determine the performance that he wishes to achieve. A filter aid can

then be selected on the basis of a performance test and be described in terms of a material specification. Alternate filter aids can then be evaluated in terms of whether they can be expected to provide equivalent performance.

The filtration performance of a filter medium is a function of many variables, some of which are physical characteristics of the medium itself. Physical characteristics of the medium that are important include particle size and shape, and the volume of the voids in the cake as it is formed on a filter septum for use in filtration.

A particle size analysis of a filter aid can be made using a variety of methods including the hydrometer method, the Coulter counter method, and the permeability method. The results of the various methods will not necessarily agree unless the particles are spherical. Any of the methods can be used for determining the particle size of diatomite but the hydrometer method cannot be used for perlite because of the variable specific gravity of particles in this type of filter aid. The permeability method is particularly appropriate for both diatomite and perlite filter aids because the same test used for determining the filter aid resistance, ξ, $[F^{-1}L]$ is used for determining the bulk density. Thus, bulk density and particle size can be determined under the same test conditions.

In order to determine the volume of voids in a filter cake, it is necessary to determine both the effective specific gravity of the media and t bulk density of the filter aid as it exists in a filter cake. The effective specific gravity of the filter media, ρ_F, is defined as the ratio of the weight of a unit volume of filter aid to the weight of an equal volume of water at 20°C and is expressed in dimensionless terms.

The bulk density of the filter media, γ_F, is defined as the weight of the filter media per unit volume of a filter cake as it would be formed in filtration. The bulk density of the filter media is usually expressed in units of pounds per cubic foot.

The bulk density, effective specific gravity, and void volume of a filter media are related, as expressed in the following equation:

$$\text{Void ratio} = \frac{\text{volume of voids in filter cake}}{\text{volume of filter cake}} = \frac{\gamma_w - (\gamma_F/\rho_F)}{\gamma_w}$$

$$= 1 - \frac{\gamma_F}{\gamma_w \rho_F} \qquad (1)$$

where γ_F = bulk density of filter aid, $[FL^{-3}]$

γ_w = density of water, $[FL^{-3}]$

ρ_F = effective specific gravity of filter media, $[FL^{-3}/FL^{-3}]$ or $[-]$*.

In order to determine how much of each potentially equivalent grade of filter aid is required to provide the same filtration resistance, an equation was developed to relate the physical characteristics of a filter aid to the resistance of the filter cake formed during filtration. This equation is in the following form:

$$\alpha = \frac{k}{V_v} \qquad (2)$$

where α = filter cake resistance index, $[L^{-2}]$

k = constant depending on the suspended solid removed by filtration

V_v = volume of voids in the filter cake = $\frac{C_F}{\gamma_F}\left(1 - \frac{\gamma_F}{\gamma_w \rho_F}\right)$, cu ft per 10^6 lb of filtrate, $[L^3F^{-1}]$

---

*[—] Dimensionless units.

C_F = body feed concentration, ppm by weight* [–]

In the filtration of an iron-bearing waters used to simulate the operation of an Erdlator, the constant in the equation was found to be a function of the hydraulic radius of the filter aids:

$$k = \frac{12.0 \times 10^9}{R_{em}}$$

where R_{em} = hydraulic radius, microns [L].

In order to determine the amount of body feed, C_F, for any other grades of filter aids needed to provide equivalent performance, it is necessary to be able to determine average values for the following characteristics for each filter aid:

- Specific gravity of the filter aid, ρ_F,
- Bulk density of the filter aid, γ_F, and
- Filter aid resistance index, the ξ-index (or β_0).

---

*[–] Dimensionless units.

TEST PROCEDURE – FOR DETERMINATION OF EFFECTIVE SPECIFIC GRAVITY OF PRECOAT FILTER AIDS

General

The following test procedure has been established to determine the effective specific gravity of filter aids for use in calculating the voids in a filter cake. Since the actual specific gravities of the individual particles of perlite filter aid vary from values below 1 (floaters) to values over 2 (sinkers), the specific gravity desired is the net or effective specific gravity of the material. In processing, perlite materials are expanded and crushed. Some particles will not be crushed and will include bubbles of gas which will provide a particle specific gravity less than 1. Others will be crushed and their specific gravity will approach the specific gravity of the raw material. The void volume available for passage of water through the filter will be determined by use of the effective specific gravity of the bulk material.

With diatomite filter aids, the particles do not generally include gas bubbles within the particles. Therefore, there are generally no floaters in diatomite filter aids. The very porous structure of the particles, however, sometimes retains some gas bubbles attached to the filter aid particles. Thus, diatomite filter aids should be soaked for a short period with mixing at some step during the determination of their effective specific gravity to eliminate attached air bubbles.

Scope

This test method is intended for use in determining the effective specific gravity of diatomite and perlite filter aids by use of a pycnometer. The effective specific gravity value obtained may be used in the determination of the effective void volume found in filter cakes formed using the filter aid. The effective specific gravity value determined using diatomite filter aids may also be used in calculation of the particle size distribution of the filter aid in connection with the hydrometer method of particle size analysis.

Definition

Specific gravity, ρ_F, is the ratio of the weight in air of a given volume of the filter aid being tested to the weight in air of an equal volume of distilled water at 20°C.

Apparatus

The apparatus required to determine the effective specific gravity of filter aids includes:

1. A Pycnometer. A standard, Class A, volumetric flask with a capacity of 500 ml may be used as the pycnometer. (Example: E. H. Sargent & Co., Flask S-34855-20, volumetric, Polyethylene stopper, Class A, Pyrex Brand Glass, Size K (500 ml), $4.95 each or $53.52/dozen.)

2. Balance. A top-loading balance with sensitivity/readibility of 0.01 g and of sufficient capacity to accommodate the flasks to be used. (Example: E. H. Sargent & Co., Balance S-2552-40, top-loading, direct reading, one-pan, 1200 g, Mettler P1200, $695.00.)

3. Thermometer. A mercury thermometer with a range of - 20°C to + 102°C graduated in 1°C subdivisions. (Example: E. H. Sargent & Co., Thermometer S-80006, B, $2.00.)

4. Stirring Apparatus. A magnetic stirrer complete with several plastic stirring bars. (Example: E. H. Sargent & Co., Stirring Apparatus S-76490, magnetic, variable-speed, Sargent, $34.00.)

5. Wash Bottle. A polyethylene wash bottle is used to add distilled water to, and to wash down, the neck of the volumetric pycnometer. (Example: Sargent & Co., Wash Bottle S-9487C, bottle, washing, Polyethylene, one piece cap, 500 ml, $0.65 each.)

Procedure

The following laboratory procedure has been developed for determining the specific gravity of filter aids:

1. Determine the weight of a clean, dry volumetric flask containing a magnetic stirring bar, recording the weight to the nearest 0.01 g. This weight is the tare weight of the volumetric flask, W_T.

2. Fill the volumetric flask three-fourths full (at least 40 g) of the sample of filter aid whose specific gravity is to be

determined. Clean the outside of the flask and weigh the flask plus the filter aid plus the magnetic stirring bar, recording the weight, W_{TS}, to the nearest 0.01 g.

3. Slowly add distilled water to the flask to wet the filter aid thoroughly and to suspend it in a slurry, while stirring the flask contents on a magnetic stirrer. The distilled water should be drawn from storage maintained at a temperature as close to 20°C as possible* to reduce variation in water temperature during the test and to eliminate the need for temperature correction. Water should be added until the flask is about half full. The removal of gases can be expedited by gently stirring the suspension in the flask on the magnetic stirrer. Then, the sample should be allowed to soak for 30 min.

4. After soaking, slowly add distilled water to the flask while the contents are being mixed to maintain a uniform suspension. When the water rises in the neck of the flask, quickly shut off the stirrer and bring the water level to the flask capacity graduation mark. Subsequently, floaters in the filter aid may rise into the neck of the flask, obscuring the capacity mark. (Some practice may be required to add the proper amount of distilled water to reach this mark quickly, especially with perlite filter aids, before the floaters obscure the capacity mark.) Dry the outside of the flask and weigh the flask plus the magnetic stirring bar plus filter aid sample plus distilled water, recording the weight, W_{TSW}, to the nearest 0.01 g.

---

*A water temperature of 20°C $\pm$ 3°C is satisfactory.

5. Record the temperature of the contents of the flask to the nearest degree Centigrade.

6. Dispose of the contents of the flask, clean and replace the magnetic stirring bar in the flask, and refill the flask with distilled water to the capacity graduation mark. The temperature of the distilled water should be the same as that used in step 3 and recorded in step 5. Weigh the flask plus the magnetic stirring bar plus the distilled water and record this weight, W_{TSW}, to the nearest 0.01 g.

7. Dispose of the contents of the flask, clean and dry the flask.

Calculations

Calculate the specific gravity of the filter aid sample from the recorded values of W_T, W_{TS}, W_{TW}, and W_{TSW}. The data required in a specific gravity determination may be recorded on a data sheet of the type shown in Exhibit 1. The specific gravity of the filter aid, based on water at a temperature of 20°C, may be calculated as follows:

$$\text{Specific gravity, } \rho_F = \frac{(W_{TS} - W_T)}{(W_{TS} - W_T) + (W_{TW} - W_{TSW})}$$

where W_T = weight of flask plus stirring bar in grams

W_{TS} = weight of flask plus stirring bar plus filter aid sample, in grams

$(W_{TS} - W_T)$ = dry weight of filter aid sample, in grams

W_{TW} = weight of flask plus stirring bar plus distilled water, in grams

Exhibit 1. SAMPLE DATA SHEET.

Data Sheet

EFFECTIVE SPECIFIC GRAVITY DETERMINATION

FOR

FILTER AIDS

Sample designation: _____ Date: _____

Volumetric flask number: _____ Technician: _____

Weight of volumetric flask plus sample, W_{TS} = _____ g*

Weight of volumetric flask, W_T = _____ g

Weight of filter aid sample, $(W_{TS} - T_T)$ = _____ g

Temperature of water when W_{TSW} is determined = _____ °C

Weight of volumetric flask plus sample plus water, W_{TSW} = _____ g

Weight of volumetric flask plus water, W_{TW} = _____ g

$(W_{TSW} - W_{TW})$ = _____ g

$$\rho_F = \frac{\text{weight of sample}}{\text{weight of water displaced}} = \frac{(W_{TS} - W_T)}{(W_{TS} - W_T) - (W_{TSW} - W_{TW})}$$

= _____

ρ_F = specific gravity = _____

*All weights should include the magnetic stirring bar in the flask.

W_{TSW} = weight of flask plus stirring bar plus filter aid sample plus distilled water, in grams

$(W_{TS} - W_T) - (W_{TW} - W_{TSW})$ = weight of water displaced by the filter aid sample, in grams.

The specific gravity should be recorded only to the nearest 0.01 unit. A sample calculation is shown in Exhibit 2.

Accuracy

Using the test procedure described above, in which the weights are determined to 0.01 g and the water temperature is held to 20°C ± 3°C, the error in determining the specific gravity should be in the order of ± 0.02. The effective specific gravity determined in this way is more precise than required when using the effective specific gravity values in filtration calculations.

This test procedure, however, ignores the corrections which may be necessary due to expansion or contraction of both the volumetric flask and the filter aid (essentially glass) due to changes in temperature. For maximum accuracy, the specific gravity determinations should be carried out with water at 20°C. Then, no temperature corrections are involved.

The level of effect of temperature on the specific gravity of a filter aid sample due to differential expansion of the water and the filter aid may be demonstrated by the following example:

Assume a filter aid whose density is 2.00 g/cm^3 at 15°C. Determine the effect of temperature on the specific gravity of the filter

Exhibit 2. SAMPLE CALCULATION.

Data Sheet

EFFECTIVE SPECIFIC GRAVITY DETERMINATION

FOR

FILTER AIDS

Sample designation: S2-7 Date: 1 April 1968

Volumetric flask number: 500-1 Technician: Shouse

Weight of volumetric flask plus sample, W_{TS}	= 200.40	g*
Weight of volumetric flask, W_T	= 162.65	g
Weight of filter aid sample, $(W_{TS} - W_T)$	= 37.75	g
Temperature of water when W_{TSW} was determined	= 20	°C
Weight of volumetric flask plus sample plus water, W_{TSW}	= 677.08	g
Weight of volumetric flask plus water, W_{TW}	= 665.40	g
$(W_{TSW} - W_{TW})$	= 11.68	g

$$\rho_F = \frac{\text{weight of sample}}{\text{weight of water displaced}} = \frac{(W_{TS} - W_T)}{(W_{TS} - W_T) - (W_{TSW} - W_{TW})}$$

$$= \frac{37.75}{37.75 - 11.68}$$

ρ_F = specific gravity

$$= \frac{37.75}{26.07}$$

$$= 1.45$$

*All weights should include the magnetic stirring bar in the flask.

aid at a temperature of 20°C. The densities of the solid and the water at the two temperatures would be:

	15°C	20°C
SOLID DENSITY:	$\frac{2.00\ g}{cm^3}$	$\frac{2.00\ g}{(1 + 0.000025 \times 5)} = \frac{2.00\ g}{1.000125\ cm^3}$

The coefficient of cubical expansion of glass ranges from 0.23 to 0.26×10^{-4}*. Since perlite and diatomite are essentially glass, they can be assumed to have a coefficient of cubical expansion of 0.25×10^{-4}, which is the increase in volume per unit volume per degree Centigrade. Thus, 1 cm^3 of glass at 15°C would occupy (1 + 0.000025 × 5) or 1.000125 cm^3 at 20°C.

WATER DENSITY: $\frac{0.999099\ g}{cm^3}$** → $\frac{0.998203\ g}{cm^3}$

15°C | 20°C

SPECIFIC GRAVITY CALCULATIONS:

$\rho = \frac{2.0000}{0.999099} = 2.0018$ No temperature correction applied

$\rho\ 20°C = \frac{2.0000}{0.998203} = 2.0036$ Including correction for change in water density

$\rho\ 20°C = \frac{2}{1.000125 \times 0.998203} = 2.0033$ Including correction for change in <u>water</u> and <u>solid</u> density.

This sample calculation indicates that:

1. The temperature correction for a temperature change of 5°C did not affect the effective specific gravity in the second decimal place.

---

*<u>Handbook of Chemistry and Physics</u>, 42nd ed., 1961, p. 2247.

**<u>Ibid</u>., Absolute density of water, g/cm^3, p. 2142.

2. The major temperature effect is on the density of the water, the temperature effect on the solid being about 14.3% of the effect on the water,
3. The temperature effect on the density of the solid tends to overcome the effect on the density of the liquid in the specific gravity calculations, and
4. Temperature effects under 3° to $5^{\circ}C$ may be ignored.

Since each weighing would only be accurate to $\pm$ 0.01 g, the weighing inaccuracy of the minimum 40 g sample, whose true specific gravity was 2.00, might result in the following values of specific gravity.

$$\rho = \frac{40.02 \text{ g}}{19.98 \text{ g}} = 2.003$$

or

$$\rho = \frac{39.98 \text{ g}}{20.02 \text{ g}} = 1.997.$$

These calculations indicate that weighings to an accuracy of 0.01 g are consistent with the temperature correction since a specific gravity variation would only show up in the third decimal place. Errors in the recorded weight and due to ignoring corrections for temperature variations under $5^{\circ}C$ will provide a specific gravity accurate to 0.01. Thus, if the standard deviation exceeds a value of 0.01, the deviations can be attributed to other reasons, principally the difficulty in reading the pycnometer capacity mark when a large number of floaters are present.

A laboratory technician made ten replicate specific gravity determinations using samples of diatomite and perlite filter aids. The average specific gravity of the sample of diatomite was 2.30 with a standard deviation of 0.005. The average specific gravity of the perlite sample

was 1.75 with a standard deviation of 0.045. This sample had the largest percentage of floaters of any filter aid tested and represents the sample in which observing the flask capacity level would be most difficult.

TEST PROCEDURE – FOR DETERMINATION OF BULK DENSITY AND FILTRATION RESISTANCE OF PRECOAT FILTER AIDS

General

The following test procedure was developed for use in the determination of the bulk density and a filter aid resistance index of filter aids.

Scope

This method was developed to provide a quantitative determination of the bulk density and the filtration resistance of various filter aids using a filtration apparatus. The system was designed for making constant-pressure filtrations through a sample of the filter aid using a flat septum. The technique that has been adopted involves the deposition of a filter cake from a suspension of the filter aid in distilled water. As the cake forms, the rate of filtration decreases with time. Time increments required to produce successive equal increments of filtrate volume are observed. When time increments divided by volume increments are plotted against cumulative filtrate volume, a straight line is formed which is predicted from basic cake filtration theory. The slope of the line is a direct function of the filter cake resistance.

The thickness of the deposited cake can then be measured with accuracy adequate for use in the determination of bulk density of the filter cake. After the thickness of the cake is measured, the cake is pushed out of the filter cell, dried and weighed. The in-place bulk density is then determined from the measured volume and weight of the filter aid sample.

Theory

Consider the relatively simple flow equation

$$q = \frac{dV}{Adt} = \frac{dP_x}{\mu \xi dW_x} \qquad (3)$$

where

q = flow rate per unit area, $[LT^{-1}]$ or ft/sec

V = volume of filtrate filtered in time t, $[L^3]$ or cu ft

A = total filter area, $[L^2]$ or sq ft

dP_x = pressure drop across a differential cake thickness dx, $[FL^{-2}]$ or lb/sq ft

μ = dynamic viscosity, $[FTL^{-2}]$ or lb sec/sq ft

ξ = cake flow resistance based on weight of cake solids, $[LF^{-1}]$ or ft/lb

dW_x = weight of cake solids per unit area of a differential cake thickness, dx, $[FL^{-2}]$ or lb/sq ft

Equation (3) simply states that the time rate of flow per unit area equals the force causing filtration, dP_x, divided by the medium resistance, $\mu\xi dW_x$.

To derive an expression for the total weight of filter cake, Eq. (3) may be integrated as follows

$$\mu\xi q \int_o^W dW_x = \int_o^{P-P_1} dP_x \qquad (4)$$

where

P = pressure at the cake surface, $[FL^{-2}]$ or lb/sq ft

P_1 = pressure required to overcome the resistance of the filter cake supporting septum, $[FL^{-2}]$ or lb/sq ft

(The term refers to all resistance other than the cake resistance. The gage pressure at the discharge side of the filter cake support is assumed zero.)

W = total weight of cake solids per unit area, $[FL^{-2}]$ or lb/sq ft.

Integration of Eq. (4) yields:

$$q\mu\xi W = (P - P_1). \tag{5}$$

The pressure P_1 can be related to the filter cake support resistance by writing the flow equation for the filter cake support as

$$q = \frac{dV}{Adt} = \frac{P_1}{\mu R_m} \tag{6}$$

where R_m is the total filter cake support resistance per unit area, $[L^{-1}]$ or 1/ft.

Substituting the value of P_1 from Eq. (6) into Eq. (5) and rearranging,

$$q\mu\xi W + q\mu R_m = P \tag{7}$$

or

$$q = \frac{dV}{Adt} = \frac{P}{\mu(\xi W + R_m)} . \tag{8}$$

The total weight of cake solids per unit of filter area, W, can be expressed in terms of the filtrate volume as

$$W = \frac{cV}{A} \tag{9}$$

where c is the weight of solids per unit volume of filtrate, $[FL^{-3}]$ or lb/cu ft.

Substituting Eq. (9) into Eq. (8) yields

$$\frac{dV}{Adt} = \frac{AP}{\mu(\xi cV + R_m A)} . \tag{10}$$

Equation (10) is in the general form of the rate filtration equation.

Rearranging Eq. (10) yields:

$$\frac{1}{dV/dt} = \frac{\mu \xi c}{A^2 P} V + \frac{\mu R_m}{AP} . \qquad (11)$$

According to Eq. (11), plotting the results of a constant rate filtration with $\frac{1}{dV/dt}$ or dt/dV vs V will give a straight line whose slope is K_2. The value of K_2 is

$$K_2 = \frac{\mu \xi c}{A^2 P} . \qquad (12)$$

In order to determine the resistance of a filter aid, a constant-pressure filtration test must be made using the filter aid in which data are collected to determine K_2. Since the viscosity of water, μ, is a function of temperature*, its value can be readily determined (Table 1). The value of filter area will be known and the value of P can be established at a standard value. The value of c can be determined and ξ can be calculated from

$$\xi = \frac{K_2 A^2 P}{\mu c} . \qquad (13)$$

Table 2 summarizes the equations required in calculation of experimental data collected for the purpose of determining both γ_F and ξ.

---

*The temperature of the water used in the test should be about the same as the temperature of the test apparatus and in the range from 15° to 25°C, if possible.

Table 1. Viscosity of water.

Water temperature °C	Water viscosity, μ centipoises	$\frac{\text{lb sec}}{\text{ft}^2}$ ($\times 10^{-5}$)
10	1.3077	2.7312
11	1.2713	2.6552
12	1.2363	2.5821
13	1.2028	2.5121
14	1.1709	2.4455
15	1.1404	2.3818
16	1.1111	2.3206
17	1.0828	2.2615
18	1.0559	2.2053
19	1.0299	2.1510
20	1.0050	2.0990
21	0.9810	2.0489
22	0.9579	2.0006
23	0.9358	1.9545
24	0.9142	1.9094
25	0.8937	1.8665
26	0.8737	1.8248
27	0.8545	1.7847
28	0.8360	1.7460
29	0.8180	1.7084
30	0.8007	1.6723
31	0.7840	1.6374
32	0.7679	1.6038
33	0.7523	1.5712
34	0.7371	1.5395
35	0.7225	1.5090
36	0.7085	1.4797
37	0.6947	1.4509
38	0.6814	1.4231
39	0.6685	1.3962
40	0.6560	1.3701

Table 2. Summary of theoretical equations used for data calculation.

Main relationship (constant-pressure filtration)

$$\frac{dt}{dV} = K_2 V + \frac{\mu Rm}{A \times P}$$

(Plot $\Delta t/\Delta V$ vs V on arithmetic coordinate paper, slope of straight line equals K_2)

V = accumulated volume of filtrate up to any time, $[L^3]$ or ft^3

t = time of filtration, [T] or sec

ξ = filter cake resistance per unit cake weight, $[LF^{-1}]$ or ft/lb

$$\xi = \frac{K_2 A^2 \Delta P}{\mu c}$$

K_2 = filtration resistance factor, $[TL^{-6}]$ or sec/ft^6 (see Fig. 6)

A = filtering surface area, $[L^2]$ or ft^2

ΔP = total pressure drop through filter cake and support, $[FL^{-2}]$ or lb/ft^2

μ = viscosity of water at temperature of test, $[FTL^{-2}]$, or $lb\ sec/ft^2$

$c = W_c/V_T$, concentration of filter aid in suspension, $[FL^3]$, lb/ft^3

V_T = volume of filtrate, $[L^3]$ or ft^3

γ_F = in-place bulk density of filter aid in cake, $[FL^{-3}]$ or lb/ft^3

$$\gamma_F = \frac{W_c}{AT_c}$$

W_c = dry weight of filter aid in cake, [F] or lbs

T_c = thickness or depth of filter cake, [L] or ft

Apparatus

Filtration Apparatus

The filtration experiments required for measuring the bulk density and cake resistance of filter aids are conducted using the constant-pressure filtration apparatus shown schematically in Fig. 1. The system

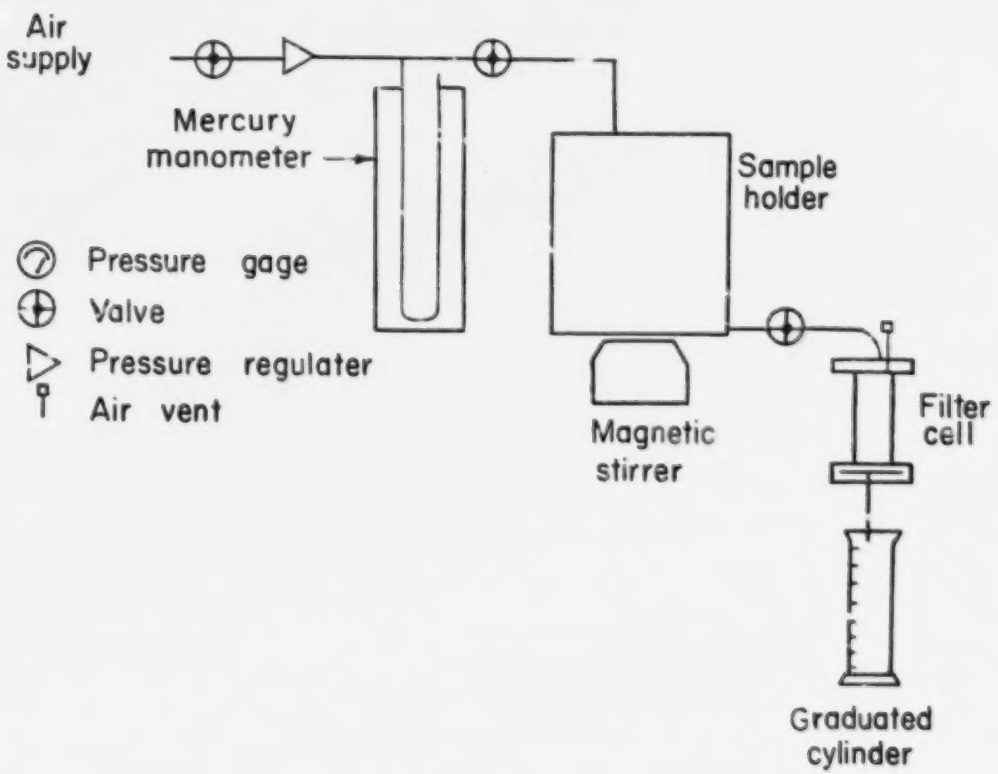


Fig. 1. Schematic diagram of bulk density and filtration resistance apparatus.

was designed for making constant-pressure filtrations using a flat filter cake support. The apparatus consists essentially of a Plexiglas sample holding tank located on top of a magnetic stirrer. A Plexiglas

filter cell fitted with a Millipore filter paper and support is connected to the sample holding tank (Fig. 1). Figures 2 and 3 show the assembly details of the sample holding tank and the filter cell. The top of the sample holding tank is connected to an air supply line to which air pressure is supplied at a pressure greater than about 50 psi. A pressure regulator capable of being controlled to provide a discharge pressure of 30 psi $\pm$ 0.1 psi should be installed in the line to the sample holding tank. The air pressure in the sample holding tank should be able to be measured using an accurate ($\pm$ 0.25 psi) pressure gage or a mercury manometer. Filtrate from the filter cell is collected in a 1000 ml graduated cylinder placed beneath the outlet pipe of the filter cell. The various essential components of the filtration apparatus include the following:

1. Air (or other gas) Pressure A source of air or other gas pressure is required. In most laboratories where air is maintained under pressure greater than 40-50 psi, a direct connection of the air line may be made through an air-pressure regulating value to the sample holding cell. The pressure regulator must be capable of reducing the line pressure to a pressure of 30 psi in the sample holder. The air pressure in the sample holder must be determined with an accuracy of about $\pm$ 1%. To accomplish this, a standard pressure gage reading accurately to 0.25 psi or a mercury manometer may be used. The specialized equipment required may be satisfied by the following items:

a) Air Pressure. Lacking line pressure in the laboratory, a portable utility compressor can be used to supply the required air pressure. (Example: W. W. Granger, Inc., 66 Washington Ave., Des Moines,

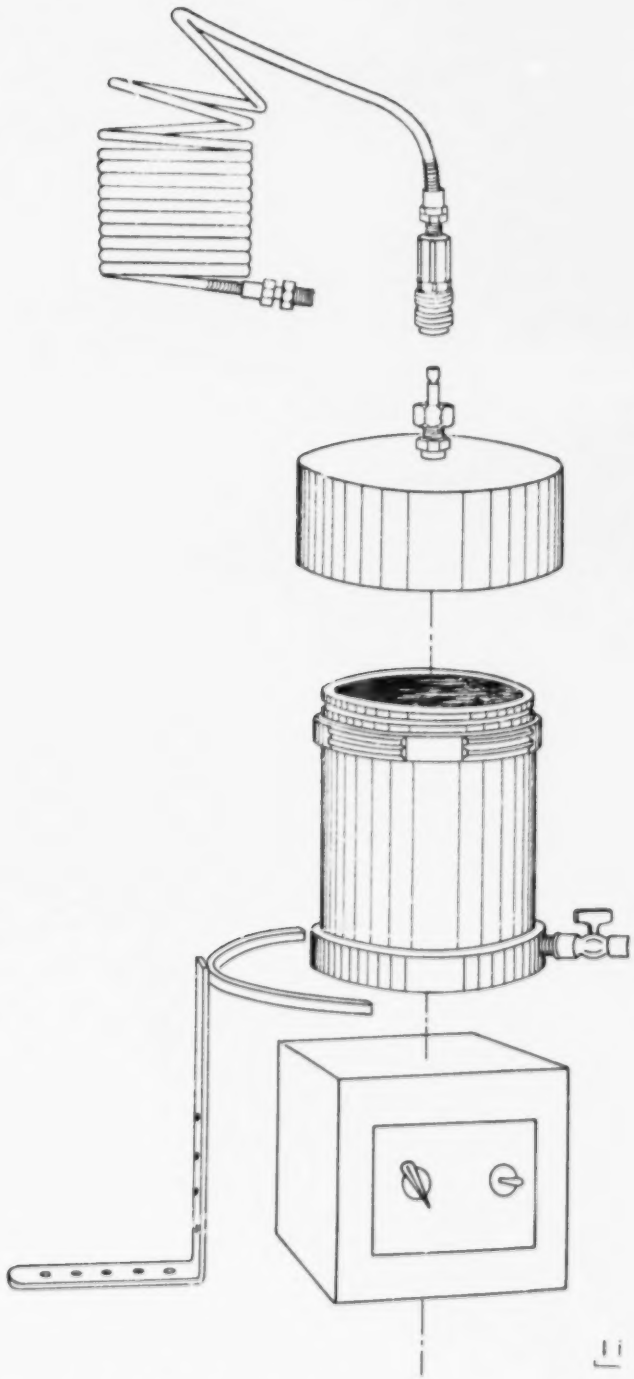


Fig. 2. Sample holder.

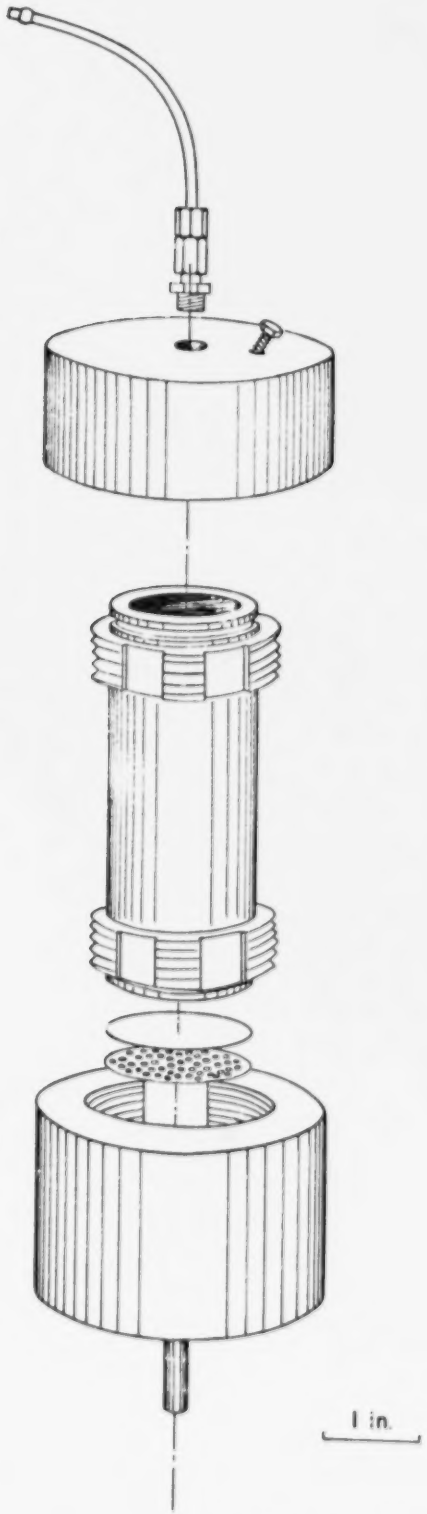


Fig. 3. Filter cell.

Iowa; ½ hp, 12-gal portable utility compressor, 1.86 CFM free air at 60 psi, No. 1Z910, $89.50.)

b) Air-Pressure Regulator. A pressure regulator will reduce line pressure to 30 psi in the sample holding tank. (Example: Fisher Governor Co., Marshalltown, Iowa, type 67 air-pressure regulator, maximum inlet, 250 psi, outlet range 0-35 psi ± 0.1 psi, $17.00.)

c) Pressure Measuring Device. A pressure gage or mercury manometer is needed to measure the filtration pressure in the sample holding tank (30 psi) to an accuracy of 1%. (Example: Meriam Instrument Co., Cleveland, Ohio, Manometer range – 100 in., Model 30EB25 WM, stainless steel construction, scale (psi and tenths), mercury, cost $113.00 or Marshalltown Manufacturing Inc., Marshalltown, Iowa; Inspectors test gage, Fig. No. 63, 0-30 psi, ¼ psi marking division, accuracy ± 0.5% of full scale reading, standard finish-brass, $37.74.)

2. Stirring Apparatus Magnetic stirrer to keep the filter aid in suspension in the sample holding tank. (Example: E. H. Sargent and Co., Stirring apparatus S-76490, magnetic, variable-speed, Sargent, $34.00.)

3. Replaceable Filter Cake Support 100 Millipore filter membranes for supporting the filter cake. Pore size – 0.45 micron, 37 mm diameter, Millipore filters are discarded after use; therefore, a supply of at least 100 membranes should be available at all times. (Example: Millipore Filter Co., MF-Millipore membranes, Type HA, 0.45 micron, white, plain, 37 mm, $18.50/100 membranes.)

4. Sample Holding Tank and Filter Cell These units are not available commercially and must be fabricated in accordance with the details shown in Figs. 4 and 5 and assembled as shown in Figs. 1, 2, and 3. A materials schedule for the complete apparatus is included in Exhibit 3.

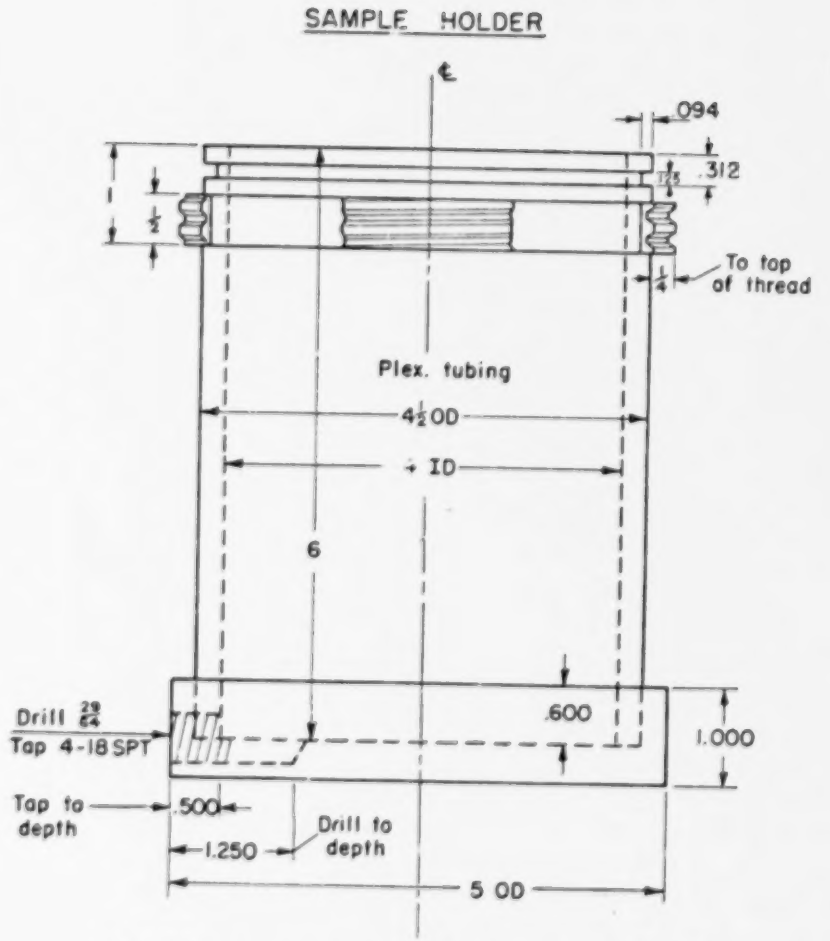


Fig. 4a. Construction drawing, sample holder.

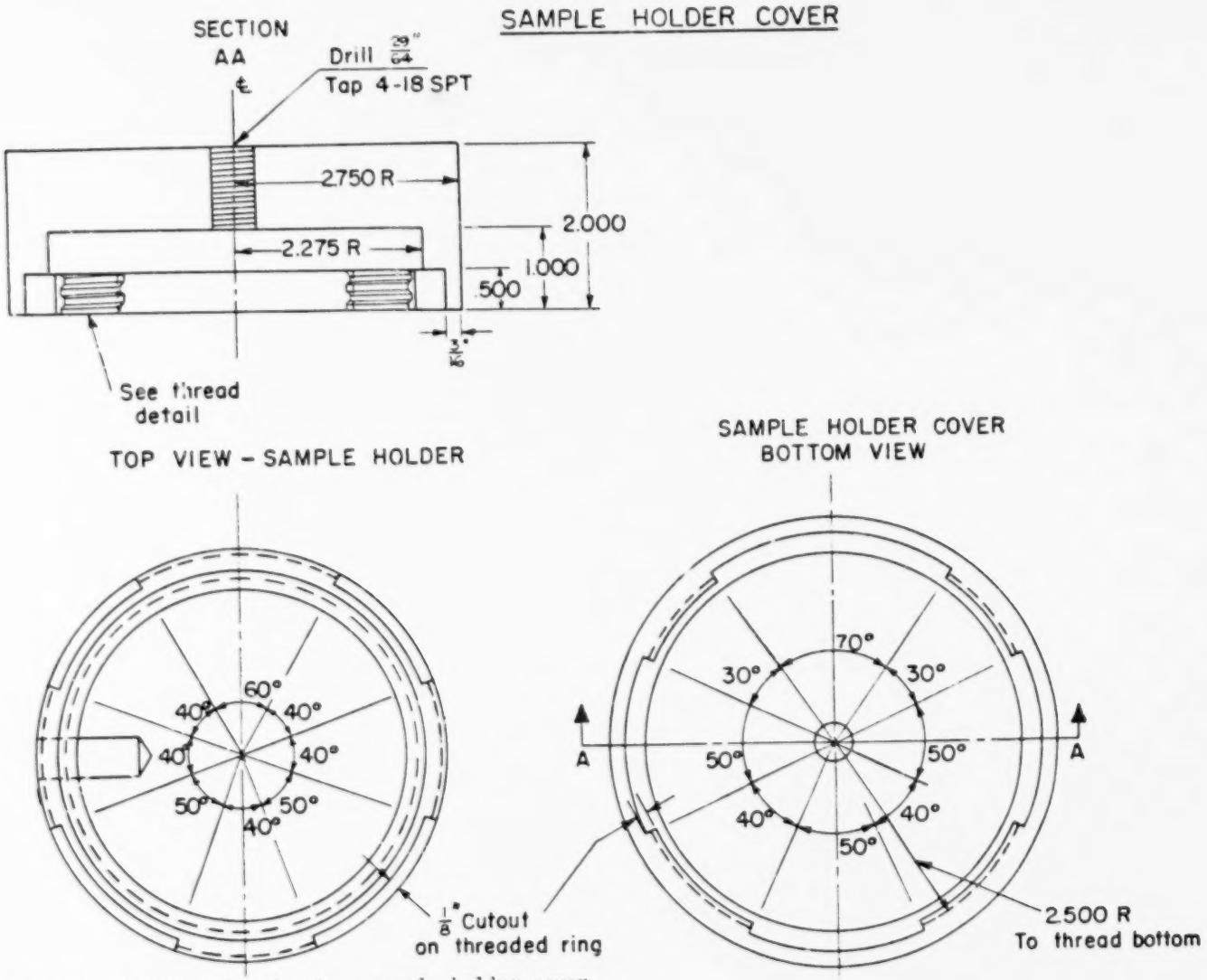


Fig. 4b. Construction drawing, sample holder cover.

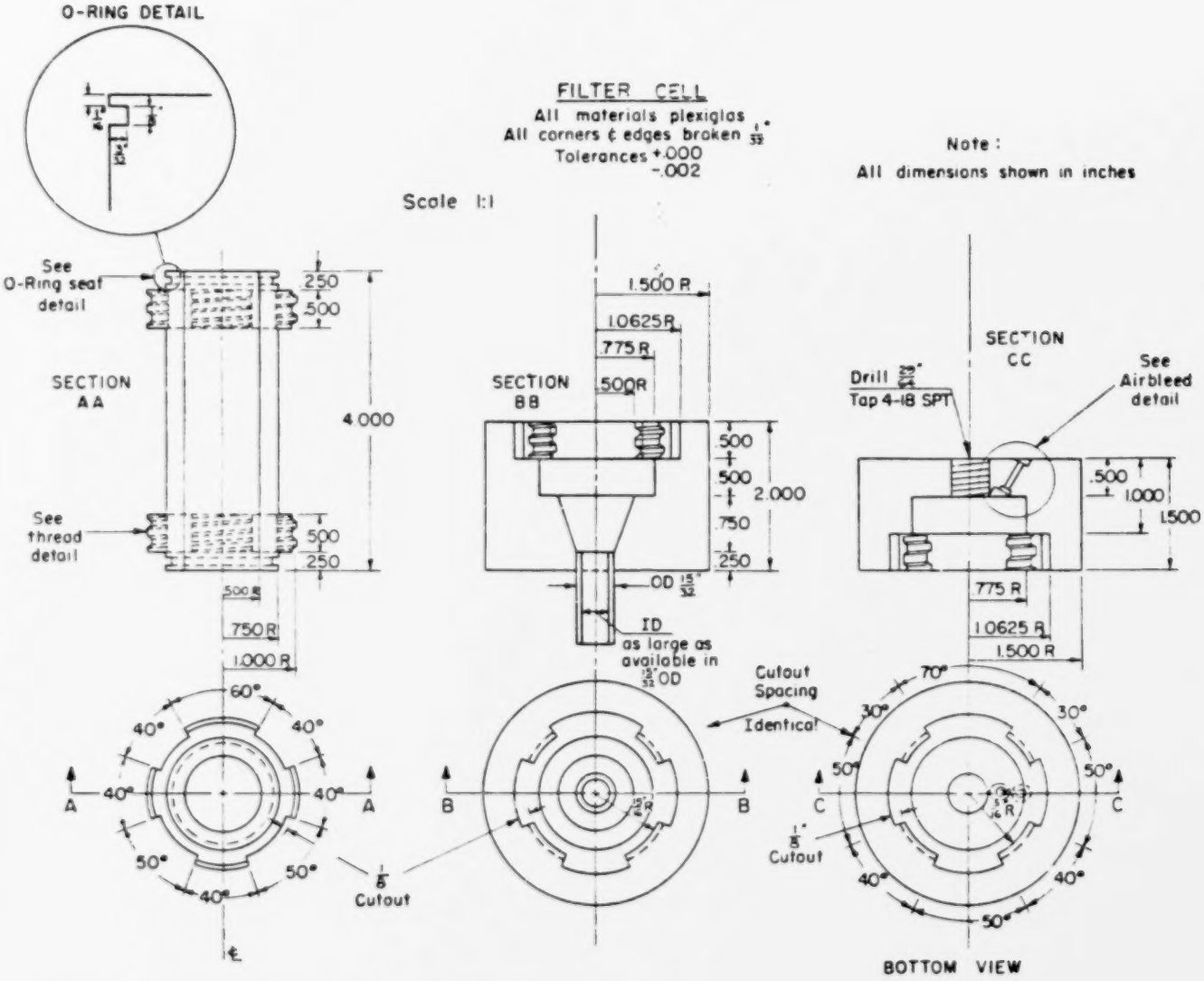
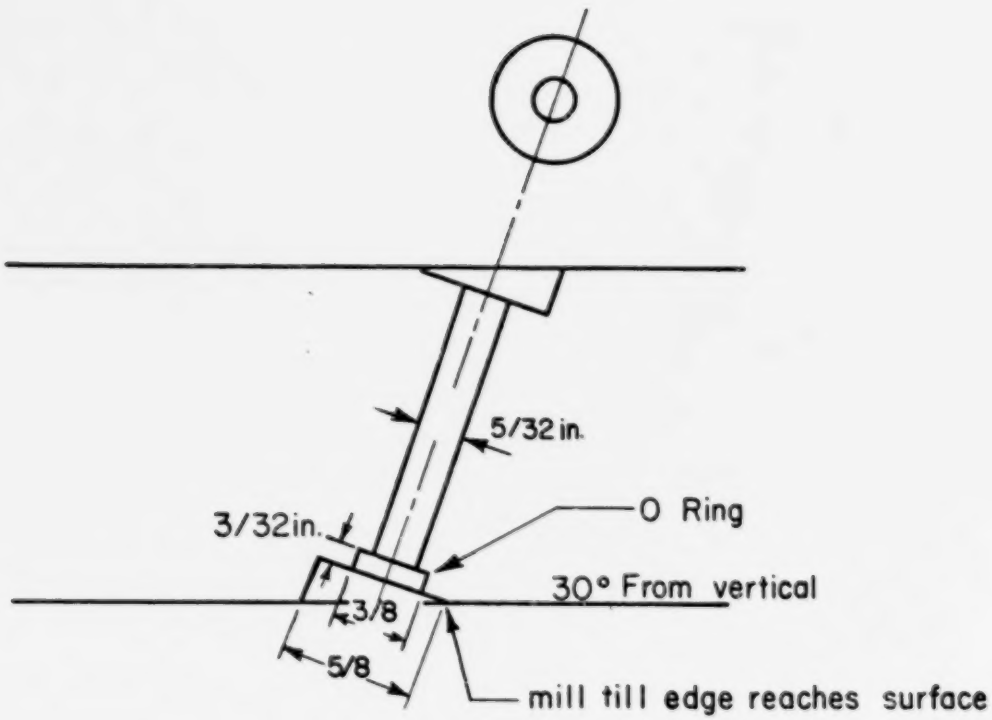


Fig. 5a. Construction drawing, filter cell.

AIR BLEED DETAIL



Valve made from

— 1 in. 6-32 flat head brass machine screw

— 1/2 in. long 1/4 in. diameter stainless compression spring

— 1/2 in. diameter 1/8 in. thick brass disk, drilled and tapped in center for 8-32

Fig. 5b. Construction drawing, filter cell air bleed detail.

5. Filtered Effluent Collection Vessel. A 1-liter graduated cylinder may be used for collecting the filtered water. (Example: E. H. Sargent & Co., S-24665J cylinder, single, graduated, Pyrex brand glass, 1000 ml, $9.19 each.)

6. Thermometer. A mercury thermometer with a range of - 20°C to + 102°C graduated in 1°C subdivisions. (Example: E. H. Sargent & Co., thermometer S-80006B, $2.00.

7. Timing Device. Two or three stopwatches or other timing devices accurate to 0.2 second are required to determine time required to filter equal increments of filtration volume. (Example: E. H. Sargent & Co., S-77446 stopwatch, 60 min, 1/5 sec, single continuous, Precision grade, $30.00 each.)

8. Filter Cake Depth Micrometer. A measuring device is required for determining the depth of the filter cake in the filter cell to an accuracy of about 0.02 to 0.03 in. (1%). A micrometer measuring lengths in inches and 64th of inches is satisfactory. (Example: Iowa Machinery and Supply Co., Inc., Lufkin depth gage, No. H511A, $5.00.)

Other Apparatus

Additional equipment or supplies required for determining bulk density and resistance of filter aids includes the following:

9. Balance. A balance sensitive to 0.01 g is required for determining the weight of filter aid in the filter cake after drying. (Example: E. H. Sargent Co., Balance S-2552-40, top-loading, direct-reading, one-pan, 1200 g, Mettler P-1200, $695.00.)

Exhibit 3.

Materials Schedule *

for

APPARATUS FOR DETERMINATION OF BULK DENSITY AND FILTRATION RESISTANCE OF FILTER AIDS

SAMPLE HOLDER

Number of Items	Description	Estimated Cost
	Cylinder, Plexiglas	
1	6" x 4½" O.D. x 4" I.D.	$ 3.48
	Sheet, Plexiglas	
1	5" x 5" x ½"	$ 1.00
1	5" x 5" x 1"	$ 1.50
1	5½" x 5½" x ½"	$ 1.25
1	5½" x 5½" x 1½"	$ 3.00
	"O" Ring	
1	4 5/8" O.D. x 4¼" I.D.	$ 0.50
	Clamp, Aluminum (hold sample holder on stirrer)	
1	15" x 4½" x 1/8"	$ 2.00
	Sub-Total	$12.73

FILTER CELL

	Cylinder, Plexiglas	
1	4" x 1½" O.D. x 1" I.D.	$ 1.00
	Sheet, Plexiglas	
2	3" x 3" x ½"	$ 0.80
2	2" x 2" x ½"	$ 0.50
	Rod, Plexiglas	
1	3" Dia x 1½"	$ 3.00
1	3" Dia x 3/4"	$ 1.50
	Tube, Plexiglas	
1	1¼" x ½" O.D. x 3/8" I.D.	$ 0.10

* McMaster-Carr Supply Co., Box 4355, Chicago 60680

Exhibit 3. (Cont.)

	"O" Ring	
2	1¼" O.D. x 1 1/8" I.D.	$ 1.00
1	5/16" O.D. x 3/16" I.D.	$ 0.18
	Wire Mesh, Stainless Steel	
1	1½" Dia, #16 Mesh, 0.023" wire dia.	$ 0.50
	Screws	
1	6-32 brass FH Machine screw and nut	$ 0.05
	Sub-Total	$ 8.63
ACCESSORIES		
	Valve, plug, brass	
1	¼"	$ 4.75
	Nipples, brass NPT	
1	¼"	$ 0.65
	Reducer, brass	
2	¼" to 1/8"	$ 1.00
	Tubing, Copper	
1	6" x ¼" O.D.	$ 0.15
	Air hose assembly	
1	¼" Retractile Nylon 10 ft long, W/¼" quick-connect coupling	$14.35
	Sub-Total	$20.90
	Total Materials Cost . .	$42.26
	Approximate Labor Requirement . .	100 hrs.

10. Oven. A 103°C drying oven is required for drying the filter cakes after they are extruded from the filter cell. (Example: E. H. Sargent & Co., S-64055 Oven, Thelco No. 14, gravity convection, electric, 150°C, single wall, $125.00.)

11. Beaker. A 250-ml beaker is required for holding the filter cakes extruded from the filter cell during drying and weighing. (Example: E. H. Sargent Co., S-4675K Beaker, Griffin, low form, with pourout, graduated, Pyrex glass, 250 ml, $0.41 each or $17.76 per case of 48.)

12. Graph paper. Arithmetic coordinate paper is required for plotting the filtration data. (Example: Keuffel and Esser Co., 10 x 10 to the centimeter, 461510, $1.95/100.)

Procedure

At the beginning of a filtration test, a filter aid suspension is prepared by adding to one liter of distilled water* the weight of filter aid under test (3 to 15 g) required to produce a cake 2.5 ± 0.2 in. deep in the filter cell. A preliminary test should be made using a known weight of the filter aid to be tested to determine the thickness of cake, T_t, that the trial weight, W_t, will produce. The weight of filter aid, W_c, required to produce a cake having a thickness of about 2.5 in. will be:

$$W_c = (2.5)\ W_t/T_t$$

The suspension is left for at least 30 min, with occasional mixing, to reach chemical equilibrium. Both diatomite and perlite filter aids, when added to water, emit Na^+ ions from their surfaces and absorb

*Prefiltered through a 0.45 micron Millipore filter.

H^+ ions from the solution, thus increasing the pH of the solution. The ion exchange process is essentially complete within 30 min.

While the filter aid suspension is soaking, a clean 0.45-micron Millipore filter membrane is installed in the filter cell and the filter cell attached to the sample holder. The air-pressure pump is put into operation and adjusted to provide a pressure of 30 psi in the filter aid sample holder. The air pressure is shut off and the filter aid suspension is introduced to the filter aid sample holder and kept under continuous agitation using the magnetic stirrer. The suspension, under atmospheric pressure, is allowed to flow into the filter cell until almost all of the air* in the filter cell is bled out by depressing the air-bleed valve (Figs. 3 and 5b).

The filtration cycle is started by again providing 30 psi of pressure in the system. The filter aid suspension will be forced to flow through the filter cell forming a cake of filter aid on the surface of the membrane (filter area, A = 0.891 $in.^2$). All of the filtrate should be collected in the one liter graduated cylinder. When exactly 100 ml of filtrate has been collected, one stopwatch should be started and the time required to filter 100 ml of suspension timed. When 200 ml of filtrate have been collected, the first stopwatch is stopped, (read and recorded) and a second stopwatch simultaneously started. Using two or more stopwatches, the times required to filter successive 100-ml volumes of filtrate can be determined, and recorded until the run is terminated. The time required to filter successive 100-ml volumes will increase progressively. The data may be recorded on data sheets of the type shown in Exhibit 4.

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*If all of the air is bled out, filter aid may foul the air bleed valve and cause leakage.

Exhibit 4. SAMPLE DATA SHEET.

Data Sheet

DETERMINATION OF IN-PLACE BULK

DENSITY AND FILTRATION RESISTANCE

Operator: _____ Date: _____

Series _____ Run No. _____

Suspension: _____

Pressure drop: _____ Filter area: _____

Filtrate temp. _____

V ml	ΔV ml	Δt sec	Δt/ΔV sec/ml	K_2 sec/ft^6	ξ ft/lb

Tc =

Wc =

γ_F =

Total volume of filtrate =

c =

Wt of beaker + sample = _____ g

Wt of beaker _____ = _____ g

Wt of sample = _____ g

After timing of filtrate volumes is concluded and the temperature of the filtrate has been measured, the system is left under the applied air pressure until all possible filtrate is forced through the filter cake. The filter cell is then disassembled and the cake thickness is measured as accurately as possible using the depth gage. An average of three to five depth readings may be used. The filter cake is then carefully pushed out of the filter cell and collected in a tared beaker, dried in the 103°C drying oven to constant weight (2-3 hrs), and weighed.

The bulk density of the filter aid is determined from the dry weight of the filter cake and its in-place volume (filter cell area x cake depth).

The filter aid weight per unit volume of filtrate, c, can be determined from the dry weight of the filter cake and the total volume of filtrate collected in the graduated cylinder. This value is needed in the determination of the filter cake resistance index, or the ξ-index.

The filter cake resistance may be determined by use of Eq. (13). A plot of the data is made in which $\Delta T/\Delta V$, in (sec/ml) is plotted on arithmetic coordinate paper as ordinate vs the volume of filtrate, V, in milliliters. A straight line is drawn through the data and its slope (sec/ml^2) is determined. In using Eq. (13), the variables must be expressed in the following units:

K_2, units of sec/ft^6

A^2, units of ft^4

ΔP, units of lb/sq ft

μ, units of lb sec/ft^2

c, units of lb/cu ft

ξ, units of ft/lb.

The following conversion factors are used to convert to the units of Eq. (13):

		To Convert	
	From	To	Multiply by
K_2	sec/ml^2	sec/ft^6	8.018×10^8
ΔP	cm of Hg	lb/ft^2	27.8
ΔP	lb/in^2	lb/ft^2	144
μ	centipoises	lb sec/ft^2 (Table 1) (kinematic viscosity)	2.08855×10^{-5}
c	g/l	lb/ft^3	6.243×10^{-2}
γ_F	g/in^3	lb/ft^3	3.81
ξ	ξ(ft/lb)	$\beta_o(ft^{-2})$	62.310×10^{-6}

In the calculations, A^2 has a standard value of 3.8285×10^{-5} ft^4.

Note: The reported values of bulk density, γ_F, and filter cake resistance, ξ, should be the averages of three separate determinations.

Calculations

Exhibit 5 shows a completed data sheet; Table 3 shows all the calculations necessary to determine bulk density and filter aid resistance for a sample of C-545 filter aid. It should be noted that the thickness of cake used in the illustrated test was less than the value of 2.5 in. specified previously. The test should have been terminated and repeated using a sample containing about 12 g of filter aid per liter:

$$W_c = (2.5)(8.13)/1.719) = 11.8 \text{ g.}$$

Figure 6 shows the graphical plot of the data required to determine K_2.

Exhibit 5. DATA FROM A CONSTANT-PRESSURE FILTER RUN

Data Sheet

DETERMINATION OF IN-PLACE BULK

DENSITY AND FILTRATION RESISTANCE

Operator: Shouse Date: 24 October 1967

Series J4-4 Run No. 2

Suspension: J4-4 in water

Pressure drop: 151.8 cm Hg Filter area: 0.891 $in.^2$

Filtrate temp. 25°C, $\mu = 1.8665 \times 10^{-5}$ $lb/sec/ft^2$

V ml	ΔV ml	Δt sec	Δt/ΔV sec/ml	K_2 sec^2/ft^6	ξ ft/lb
100					
200	100	6.6	.006		
300	100	8.3	.083		
400	100	9.0	.090	1.153×10^5	2.17×10^9
500	100	11.0	.110		
600	100	12.0	.120		
700	100	13.8	.138		
800	100	15.3	.153		
900	100	16.7	.167		
1000	100	18.0	.180		

Tc = 1.719 in.*

Wc = 8.13 g

$\gamma_F = 20.19$ lb/ft^3

Total volume of filtrate = 1104 ml or 1.104 liter

c = .46 lb/ft^3

Wt beaker + sample = 68.23 g

Tare wt beaker # 3 = 60.10 g

Wt of sample = 8.13 g

*Note: This thickness is less than the 2.5 in. thickness specified.

Table 3. Reduction of data from Figs. 4 and 5.

$$\gamma_F = \frac{W_c}{A \times T_c} = \frac{1.79 \times 10^{-2}}{6.188 \times 10^{-3} \times 0.1433} = 20.19 \text{ lb/ft}^3$$

$W_c = 8.13$ g
$= 8.13 \text{ g}/(453.59 \text{ g/lb})$
$= 1.79 \times 10^{-2}$ lb

$T_c = 1.719$ in.
$= 1.72 \text{ in.}/(12 \text{ in./ft})$
$= 0.1433$ ft

$A = 0.891 \text{ in.}^2$
$= \frac{0.891 \text{ in.}^2}{(144 \text{ in.}^2/\text{ft}^2)}$
$= 6.188 \times 10^{-3} \text{ ft}^2$

$$\xi = \frac{K_2 A^2 \Delta P}{\mu c} = \frac{1.153 \times 10^5 \times 3.829 \times 10^{-5} \times 4226}{1.8665 \times 10^{-5} \times 0.460}$$
$$= 2.174 \times 10^9 \text{ ft/lb}$$

$K_2 = *1.4375 \text{ sec/ml}^2$ (from slope of line in Fig. 10)
$= 1.4375 \times 10^{-4} \times 8.018 \times 10^8$
$= 1.153 \times 10^5 \text{ sec/ft}^6$

$A^2 = (0.891)^2 (1/144)^2$
$= 3.829 \times 10^{-5} \text{ ft}^4$

$\mu_{25^\circ C} = 1.8665 \times 10^{-5}$ lb sec/ft^2 (Table 1)

$\Delta P = 152$ cm Hg
$= 152 \times 27.8$
$= 4226 \text{ lb/ft}^2$

$$c = \frac{W_c}{V_T} = \frac{8.13 \text{ g}}{1.104 \text{ liter}} = 7.364 \text{ g/liter}$$
$$= 7.364 \ (6.243 \times 10^{-2})$$
$$= 0.460 \text{ lb/ft}^3$$

*1.436666×10^{-4} sec/ml^2 from computer least squares analysis.

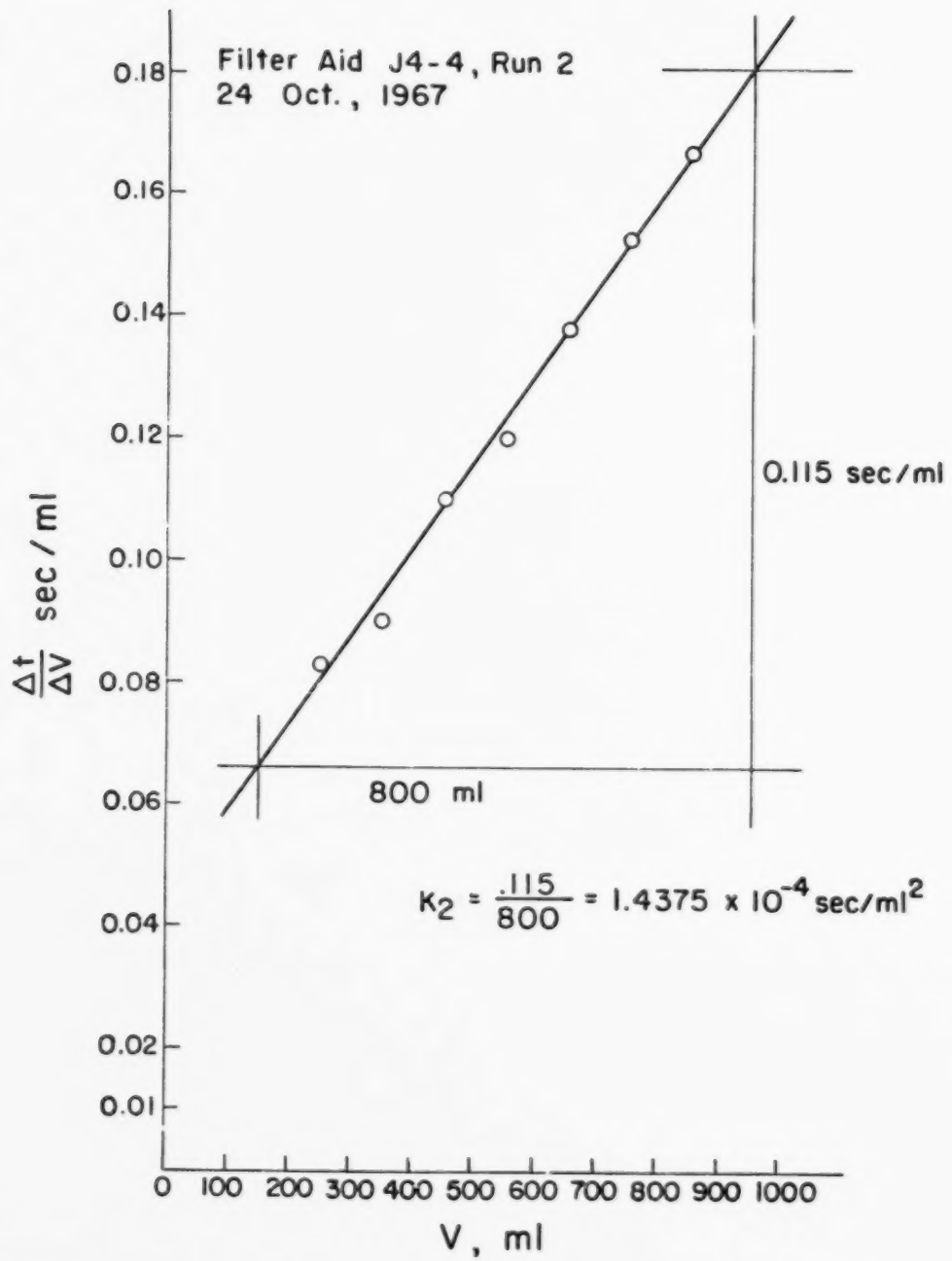

Fig. 6. Determination of K_2 from data in Exhibit 5.

Accuracy

Preliminary investigations made to determine the effect of pressure on bulk density measurements indicate that at pressure drops greater than 5 psi, the bulk density is relatively independent of the applied pressure. Filtration under pressures greater than 20 psi, however, produced smoother filter cakes. Furthermore, the problem of settling of large particles which was apparent at low pressures was reduced or eliminated in experiments conducted at higher pressure drops. In view of these observations, a pressure drop of 30 psi was adopted and is used as the standard applied pressure.

The test procedure described will provide a value for bulk density and filtration resistance index that is accurate within $\pm$ 5 percent. Twelve determinations were made of β_o and γ_F of a single sample of diatomite filter aid (E5-37). At the 95 percent confidence level, the test results were:

$$\gamma_F = 22.5 \text{ lb/ft}^3 \quad (E^*_{95\%} = 0.6)$$

$$\beta_o = 1.58 \times 10^5 \text{ ft}^{-2} \quad (E_{95\%} = 0.13 \times 10^5)$$

The results indicate that a single determination should provide a γ_F result within 3 percent of the mean value and a β_o result within 9 percent of the mean value. If the average of three determinations is used, the values of γ_F and β_o should be within 2 and 5 percent, respectively, of the mean value.

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*$E_{95\%}$ = 2 x standard deviation.

ACKNOWLEDGMENT

This work was supported by the Engineering Research Institute, Iowa State University, in part through funds made available by the U.S. Army Engineer Research and Development Laboratories through Research Contract DA A K02-67-C-0408 and by the Federal Water Pollution Control Administration through FWPCA Research Grant WP-00196 and Research Fellowship 5-F1-WP-26,104 (H. R. Bridges).

The authors wish to express their appreciation to John Shouse (Research Technician), Harold Bridges (FWPCA Research Fellow), Madan L. Arora (Research Graduate Assistant), Jerry Yuan-cheng Huang (Research Assistant), and to the undergraduate and graduate students who contributed much to the success of this study.